# STIC Search Report
## EIC 1700

## STIC Database Tracking Number: 120400

| TO: | From: |
|---|---|
| Ben Sackey<br>Location: REMSEN 5B31<br>Art Unit : 1626<br>April 29, 2004<br><br>Case Serial Number: 10/088276 | Kathleen Fuller<br>Location: EIC 1700<br>REMSEN 4B28<br>Phone: 571/272-2505<br>Kathleen.Fuller@uspto.gov |

## Search Notes

Miss Fuller

Access DB# 120400

# SEARCH REQUEST FORM

**Scientific and Technical Information Center**

Requester's Full Name: BEN SACKEY Examiner #: 73489 Date: 4/26/04
Art Unit: 1626 Phone Number ~~30~~ 2-0704 Serial Number: 10/088,276
Mail Box and Bldg/Room Location: REM 5B31 Results Format Preferred (circle): PAPER DISK E-MAIL

**If more than one search is submitted, please prioritize searches in order of need.**

**********

Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: Process for preparing Fused Pyrroles

Inventors (please provide full names): Makoto Tokunaga and Yasuo Wakatsuki

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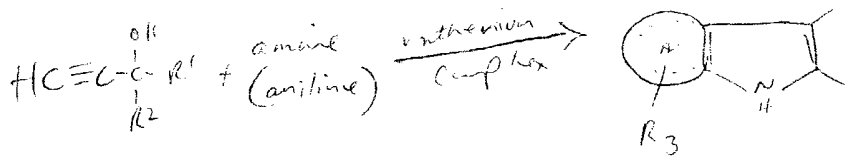
Earliest Priority Filing Date: 7/17/00

***For Sequence Searches Only* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.**

Process for preparing Condensed Pyrroles Comprising: reacting, alkyne alcohol of formula(4) with an aromatic primary amine in the presence of ruthenium catalyst complex.




claim 11
Catalyst is ruthenium salt

**********

**STAFF USE ONLY**

Searcher: K. Fuller
Searcher Phone #:
Searcher Location:
Date Searcher Picked Up:
Date Completed: 4/29/04
Searcher Prep & Review Time: 30
Clerical Prep Time:
Online Time: 28

PTO-1590 (8-01) Caveat

**Type of Search**

NA Sequence (#)
AA Sequence (#)
Structure (#) 4
Bibliographic
Litigation
Fulltext
Patent Family
Other

**Vendors and cost where applicable**

STN ✓
Dialog
Questel/Orbit
Dr,Link
Lexis/Nexis
Sequence Systems
WWW/Internet
Other (specify)

=> FILE CASRE
FILE 'CASREACT' ENTERED AT 14:00:40 ON 29 APR 2004


FILE CONTENT:1840 - 25 Apr 2004 VOL 140 ISS 17

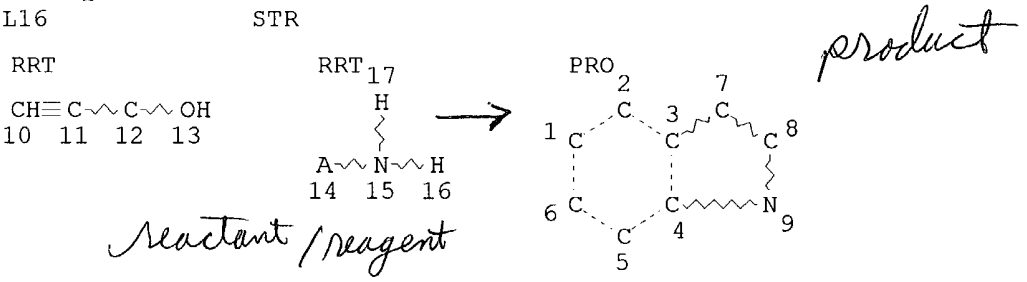
Some records from 1974 to 1991 are derived from the ZIC/VINITI data file and provided by InfoChem and some records are produced using some INPI data from the period prior to 1986.

This file contains CAS Registry Numbers for easy and accurate substance identification.

Crossover limits have been increased. See HELP RNCROSSOVER for details.

Structure search limits have been raised. See HELP SLIMIT for the new, higher limits.

=> D QUE
L16 STR




NODE ATTRIBUTES:
NSPEC IS RC AT 14
CONNECT IS E2 RC AT 9
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 17

STEREO ATTRIBUTES: NONE

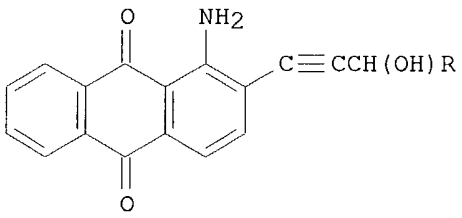
******MAPPINGS******

| NOD | SYM | ROL | NOD | SYM | ROL |
|---|---|---|---|---|---|
| 9 | N | PRO | 15 | N | RRT |
| 15 | N | RRT | 9 | N | PRO |

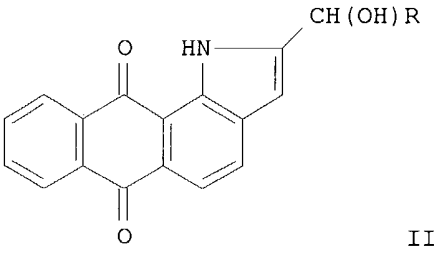
L18 7 SEA FILE=CASREACT SSS FUL L16 ( 50 REACTIONS)

=> D L18 BIB ABS IND FCRD

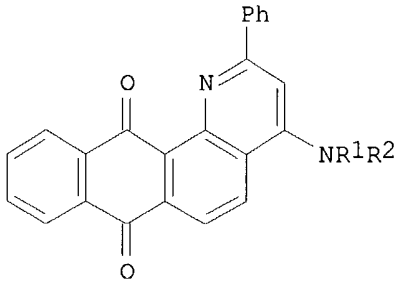
L18 ANSWER 1 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 136:385917 CASREACT
TI Transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in the presence of amines
AU Barabanov, I. I.; Fedenok, L. G.; Polyakov, N. E.; Shvartsberg, M. S.
CS Institute of Chemical Kinetics and Combustion, Siberian Branch of the Russian Academy of Sciences, Novosibirsk, 630090, Russia
SO Russian Chemical Bulletin (Translation of Izvestiya Akademii Nauk, Seriya Khimicheskaya) (2001), 50(9), 1663-1667
CODEN: RCBUEY; ISSN: 1066-5285
PB Kluwer Academic/Consultants Bureau
DT Journal
LA English
GI




I




II




III

AB When heated in piperidine, 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones (I; R = H, Pr) undergo cyclization to 2-(1-hydroxyalkyl)naphth[2,3-g]indole-6,11-diones (II). In contrast, I (R = Ph) reacts with primary and secondary amines to give 1-amino-2-(1-amino-2-benzoylvinyl)-9,10-anthraquinones, which undergo cyclization to naphtho[2,3-h]quinoline-7,12-diones (III; NR1R2 = piperidino, NHBu, NEt2). Heating I (R = Ph) with Et3N causes its dehydrogenation and isomerization.
CC 25-18 (Benzene, Its Derivatives, and Condensed Benzenoid Compounds)
ST anthraquinone amino hydroxyalkynyl cyclization; aminoanthraquinone aminobenzoylvinyl prepn cyclization; naphthindoledione hydroxyalkyl prepn; naphthoquinolinedione amino prepn
IT Dehydrogenation
Isomerization
(of amino(hydroxyphenylpropynyl)anthraquinone)
IT Heterocyclization
(transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in presence of amines)
IT Amines, reactions

RL: RCT (Reactant); RACT (Reactant or reagent)
(transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in presence of amines)

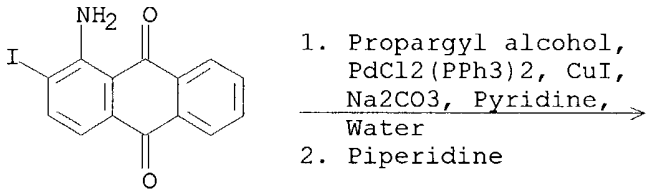
IT 768-03-6, Phenylvinyl ketone
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction with aminoiodoanthraquinone)

IT 107-19-7, Propargyl alcohol 81050-51-3
RL: RCT (Reactant); RACT (Reactant or reagent)
(transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in presence of amines)

IT 130082-03-0P 143077-30-9P 180689-19-4P 335379-02-7P 426816-32-2P
426816-33-3P 426816-37-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in presence of amines)

IT 129995-13-7P 130082-01-8P 143077-32-1P 157439-79-7P 180689-26-3P
426816-34-4P 426816-35-5P 426816-36-6P 426816-38-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(transformations of 1-amino-2-(3-hydroxyalk-1-ynyl)-9,10-anthraquinones in presence of amines)

RX(18) OF 34 - 2 STEPS




58%

RE.CNT 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> D L18 BIB ABS IND FCRD 2-7

L18 ANSWER 2 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 136:118383 CASREACT
TI Processes for preparation of indole derivatives
IN Tokunaga, Makoto; Wakatsuki, Yasuo
PA Japan Science and Technology Corporation, Japan; Riken Corp.
SO PCT Int. Appl., 27 pp.
CODEN: PIXXD2
DT Patent
LA Japanese

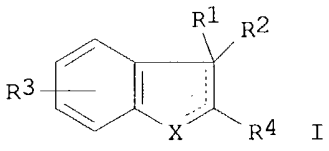
applicants

FAN.CNT 1

| | PATENT NO. | KIND | DATE | APPLICATION NO. | DATE |
|---|---|---|---|---|---|
| PI | WO 2002006226 | A1 | 20020124 | WO 2001-JP5691 | 20010702 |
| | W: US | | | | |
| | RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR | | | | |
| | JP 2002030069 | A2 | 20020129 | JP 2000-216457 | 20000717 |
| | EP 1302459 | A1 | 20030416 | EP 2001-945742 | 20010702 |
| | R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY, TR | | | | |
| | US 2004049054 | A1 | 20040311 | US 2002-88276 | 20021022 |
| PRAI | JP 2000-216457 | 20000717 | | | |
| | WO 2001-JP5691 | 20010702 | | | |

OS MARPAT 136:118383

GI



I

AB The invention provides processes for the preparation of fused pyrroles, preferably indoles, which permit the use of inexpensive aromatic amines themselves as the raw material and attain high atomic efficiency and high regioselectivity. Specifically, a process for the preparation of fused pyrroles, e.g., indoles [I; R1 = CH3, H, C6H5, CH3CH2, CH3(CH2)2; R2 = H, CH3, alkyl, aryl, electron pair; R1R2 = alkylene; R3 = H, 3-HO, 4-CH3O, 3,4-(CH3O)2, 4-CH3, 2-CH3, 4-Cl, 2-CH3OCO; R4 = H, CH3, C6H5, CH3CH2, CH3(CH2)2; X = N, NH; dotted bond = single, double] characterized by reacting an alkynol, HCCCHR1OH with an aromatic primary amine, R3C6H4NH2 in the presence of a ruthenium complex (Ru3(CO)12), more preferably with an acid or an ammonium salt (NH4·PF6). Thus, the title compound I (R1 = H; R2 = electron pair; R3 = H; R4 = (CH2)4CH3; X = NH; single bond at XCH; double at CH:CH) was prepared from CH3(CH2)4CHOHCCH and C6H5NH2 in the presence of Ru3(CO)12.

IC ICM C07D209-08
ICS C07D209-96

CC 27-11 (Heterocyclic Compounds (One Hetero Atom))

ST indole prepn catalysis ruthenium carbonyl complex catalyst

IT Catalysis
Catalysts
Regiochemistry
(processes for preparation of indole derivs.)

IT 15243-33-1, Triruthenium dodecacarbonyl
RL: CAT (Catalyst use); USES (Uses)
(processes for preparation of indole derivs.)

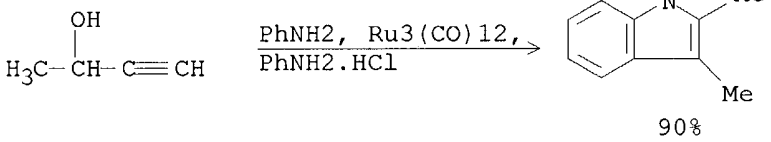
IT 62-53-3, Aniline, reactions 78-27-3, 1-Ethynyl-1-cyclohexanol 95-53-4, 2-Methylaniline, reactions 104-94-9, 4-Methoxyaniline 105-31-7, 1-Hexyn-3-ol 106-47-8, 4-Chloroaniline, reactions 106-49-0, 4-Methylaniline, reactions 134-20-3, 2-Methoxycarbonylaniline 134-32-7, 1-Naphthylamine 142-04-1, Aniline hydrochloride 591-27-5, 3-Hydroxyaniline 818-72-4, 1-Octyn-3-ol 2028-63-9, 3-Butyn-2-ol 4187-86-4, 1-Pentyn-3-ol 4187-87-5 6315-89-5, 3,4-Dimethoxyaniline

RL: RCT (Reactant); RACT (Reactant or reagent)
(processes for preparation of indole derivs.)

IT 107-21-1, Ethylene glycol, reactions 16941-11-0, Ammonium hexafluorophosphate
RL: RGT (Reagent); RACT (Reactant or reagent)
(processes for preparation of indole derivs.)

IT 91-55-4P, 2,3-Dimethylindole 828-94-4P 4757-69-1P 10257-92-8P 13141-50-9P 19013-49-1P 21296-93-5P 27505-78-8P 36729-21-2P 36729-23-4P 73177-34-1P 89188-94-3P 105908-32-5P 391611-81-7P 391611-82-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(processes for preparation of indole derivs.)

RX(1) OF 13




90%

NOTE: 120.degree., 12 h, regioselective

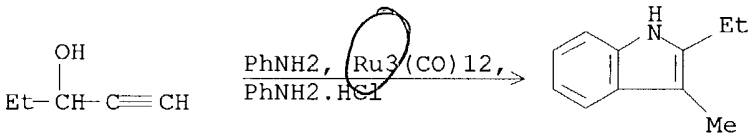
RE.CNT 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 3 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 135:166752 CASREACT
TI A practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines
AU Tokunaga, M.; Ota, M.; Haga, M.-a.; Wakatsuki, Y. applicants
CS PRESTO, Japan Science and Technology Corporation (JST), Saitama, 332-0012, Japan
SO Tetrahedron Letters (2001), 42(23), 3865-3868 2
CODEN: TELEAY; ISSN: 0040-4039
PB Elsevier Science Ltd.
DT Journal
LA English
AB 2-Substituted 3-methylindoles are synthesized with good regioselectivity from readily available substrates and catalysts, i.e., the reaction of anilines with propargyl alcs. in the presence of 0.36-1 mol % Ru3(CO)12.
CC 27-11 (Heterocyclic Compounds (One Hetero Atom))
ST indole disubstituted deriv one pot prepn; aniline reaction propargyl alc ruthenium carbonyl
IT Cyclization
(practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines)
IT Cyclization catalysts
(triruthenium dodecacarbonyl for practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines)
IT 15243-33-1, Triruthenium dodecacarbonyl
RL: CAT (Catalyst use); USES (Uses)
(practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines)
IT 62-53-3, Aniline, reactions 95-53-4, o-Toluidine, reactions 104-94-9,

p-Anisidine 106-47-8, 4-Chloroaniline, reactions 106-49-0, p-Toluidine, reactions 134-20-3, Methyl anthranilate 134-32-7, 1-Naphthylamine 142-04-1, Aniline hydrochloride 540-23-8, p-Toluidine hydrochloride 818-72-4, 1-Octyn-3-ol 2028-63-9, 3-Butyn-2-ol 4187-86-4, 1-Pentyn-3-ol 4187-87-5 16941-11-0, Ammonium hexafluorophosphate 20265-97-8, p-Anisidine hydrochloride 21436-98-6, 2,6-Dimethylaniline hydrochloride 353746-92-6 353746-93-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines)

IT 91-55-4P, 2,3-Dimethylindole 828-94-4P 10257-92-8P 19013-49-1P, 2-Ethyl-3-methylindole 21296-93-5P 27505-78-8P 36729-21-2P 73177-34-1P 89188-94-3P 105908-32-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(practical one-pot synthesis of 2,3-disubstituted indoles from unactivated anilines)

RX(1) OF 10




95%

NOTE: regioselective, no solvent, reaction run in open air, optimization study, optimized on catalyst

RE.CNT 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 4 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 130:153230 CASREACT
TI Synthesis of 2-dienylindole, SB 242784, by a three-component palladium-catalyzed coupling reaction
AU Yu, Marvin S.; Lopez De Leon, Lewilynn; McGguire, Michael A.; Botha, Glen
CS SmithKline Beecham Pharmaceuticals, King of Prussia, PA, 19403, USA
SO Tetrahedron Letters (1998), 39(51), 9347-9350
CODEN: TELEAY; ISSN: 0040-4039
PB Elsevier Science Ltd.
DT Journal
LA English
AB The synthesis of SB 242784 using a novel one-pot Castro-Stephens-Suzuki reaction as the key reaction is described.
CC 21-2 (General Organic Chemistry)
Section cross-reference(s): 27
ST SB 242784 prepn Castro Stephens Suzuki; pentadienamide chloroindolylmethoxy pentamethylpiperidinyl prepn SB 242784
IT Coupling reaction
(Castro-Stephens-Suzuki; preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)
IT Suzuki coupling reaction
(preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)
IT Alkenynes

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of enynes by three-component palladium-catalyzed coupling reaction)

IT 220185-65-9P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

IT 52670-38-9, 2-Ethynylaniline
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of (alkenyl)indoles by three-component palladium-catalyzed coupling reaction)

IT 124643-50-1P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of (alkenyl)indoles by three-component palladium-catalyzed coupling reaction)

IT 29475-88-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of (alkenyl)indoles by three-component palladium-catalyzed coupling reaction)

IT 13965-03-2, Dichlorobis(triphenylphosphine)palladium
RL: CAT (Catalyst use); USES (Uses)
(preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)

IT 74-86-2, Acetylene, reactions 95-76-1, 3,4-Dichloroaniline 115-19-5, 2-Methyl-3-butyn-2-ol 40327-96-6 81371-76-8, Methyl 3-bromo-2,2-dimethoxypropanoate
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)

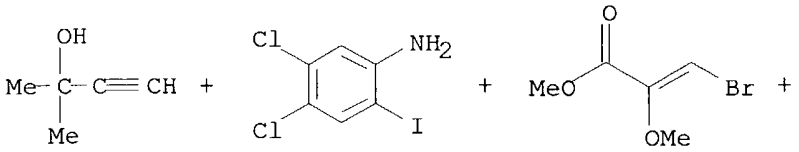
IT 119441-89-3P 180867-78-1P 220185-63-7P 220185-64-8P 220185-66-0P 220185-67-1P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)

IT 201929-00-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of SB 242784 by three-component palladium-catalyzed coupling reaction)

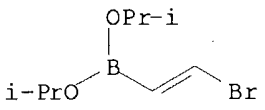
IT 764-93-2, 1-Decyne
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of enynes by three-component palladium-catalyzed coupling reaction)

IT 220185-68-2P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of enynes by three-component palladium-catalyzed coupling reaction)

RX(25) OF 38 - 3 STEPS

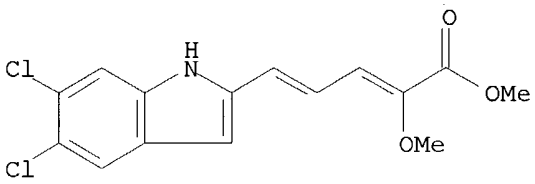


(step 2.2)



1.1. PdCl2(PPh3)2, CuI, Et3N
1.2. NaOBu-t, t-BuOH
2.1. PdCl2(PPh3)2, CuI, Et3N, THF
2.2. CsF, Water, Me2CO
3. PdCl2(MeCN)2, DMF

RX(25) OF 38 - 3 STEPS



90%

NOTE: 2) stereoselective

RE.CNT 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 5 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 130:13931 CASREACT
TI Synthesis of α-carbolines and β-carbolinones via intramolecular Diels-Alder reactions of 2(1H)-pyrazinones
AU Tahri, Abdellah; Buysens, Kris J.; Van Der Eycken, Erik V.; Vandenberghe, Didier M.; Hoornaert, Georges J.
CS Laboratorium voor Organische Synthese Department of Chemistry, K.U. Leuven Celestijnenlaan, Louvain, B-3001, Belg.
SO Tetrahedron (1998), 54(43), 13211-13226
CODEN: TETRAB; ISSN: 0040-4020
PB Elsevier Science Ltd.
DT Journal
LA English
AB 2(1H)-Pyrazinones bearing a 2-XC6H4C.tplbond.CR moiety (X = NH, NAc) are shown to undergo an intramol. cycloaddn.-elimination reaction on thermolysis in refluxing bromobenzene yielding α-carbolines or β-carbolinones. The product distribution depends strongly on the substitution pattern of the pyrazinone precursors and the solvent used for thermolysis. A high yield and selective formation of β-carbolinones is possible when heating in tetrahydronaphthalene at reflux. Use of acetic anhydride as solvent facilitated the reaction and made it possible

to realize a carbolin(on)e inaccessible by thermolysis in the previously mentioned solvents.

CC 28-2 (Heterocyclic Compounds (More Than One Hetero Atom))

ST carboline prepn; carbolinone prepn; Diels Alder intramol pyrazinone ethynylanilino deriv

IT Diels-Alder reaction
(intramol.; α-carbolines and β-carbolinones via intramol. Diels-Alder reactions of 2(1H)-pyrazinones)

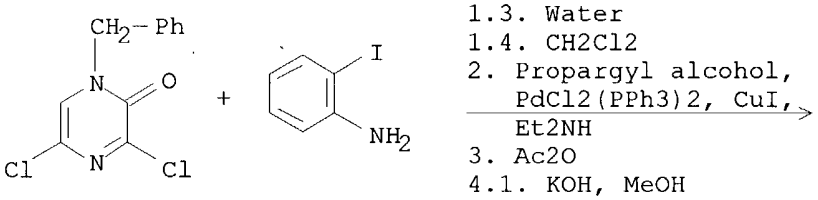
IT Elimination reaction
(α-carbolines and β-carbolinones via intramol. Diels-Alder/elimination reactions of 2(1H)-pyrazinones)

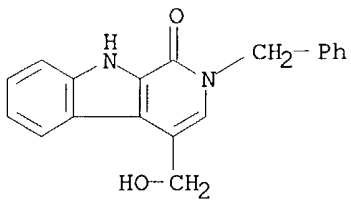
IT 107-19-7, Propargyl alcohol 536-74-3, Phenylacetylene 615-43-0, 2-Iodoaniline 1066-54-2, Ethynyltrimethylsilane 87486-35-9 87486-37-1 138610-71-6 173200-35-6 173200-36-7 216105-10-1 216105-11-2 216105-12-3
RL: RCT (Reactant); RACT (Reactant or reagent)
(α-carbolines and β-carbolinones via intramol. Diels-Alder reactions of 2(1H)-pyrazinones)

IT 162272-99-3P 216104-70-0P 216104-72-2P 216104-74-4P 216104-75-5P 216104-76-6P 216104-77-7P 216104-78-8P 216104-79-9P 216104-80-2P 216104-81-3P 216104-82-4P 216104-83-5P 216104-84-6P 216104-85-7P 216104-86-8P 216104-87-9P 216104-88-0P 216104-89-1P 216105-03-2P 216105-05-4P 216105-06-5P 216105-07-6P 216105-09-8P 216105-16-7P 216105-17-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(α-carbolines and β-carbolinones via intramol. Diels-Alder reactions of 2(1H)-pyrazinones)

IT 19839-52-2P 216104-90-4P 216104-91-5P 216104-92-6P 216104-93-7P 216104-94-8P 216104-95-9P 216104-96-0P 216104-97-1P 216104-98-2P 216104-99-3P 216105-00-9P 216105-01-0P 216105-02-1P 216105-04-3P 216105-08-7P 216105-13-4P 216105-14-5P 216105-15-6P 216105-18-9P
RL: SPN (Synthetic preparation); PREP (Preparation)
(α-carbolines and β-carbolinones via intramol. Diels-Alder reactions of 2(1H)-pyrazinones)

RX(117) OF 126 - 4 STEPS

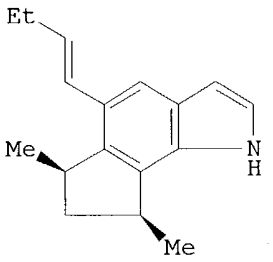


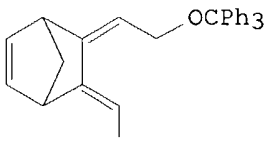

70%

RE.CNT 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

L18 ANSWER 6 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 113:6652 CASREACT
TI The total synthesis of (±)-cis-trikentrin B via allene intramolecular cycloaddition
AU Yasukouchi, Takanori; Kanematsu, Ken
CS Fac. Pharm. Sci., Kyushu Univ., Fukuoka, 812, Japan
SO Tetrahedron Letters (1989), 30(47), 6559-62
CODEN: TELEAY; ISSN: 0040-4039
DT Journal
LA English
GI




I

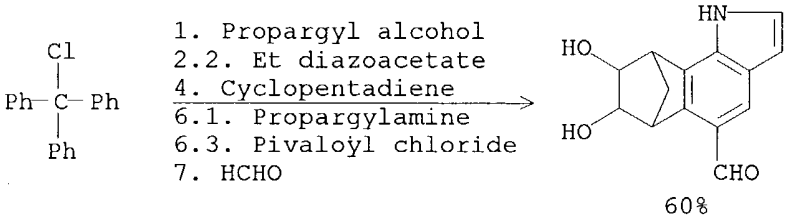



$Me_3CCONCH_2CH=C=CH_2$ II

AB The first total synthesis of (±)-cis-trikentrin B (I) based on a new indole synthesis via the intramol. Diels-Alder reaction of 1,2,3-trisubstituted allenic dienamide II is described.
CC 31-3 (Alkaloids)
ST trikentrin B total synthesis; allenic dienamide intramol Diels Alder
IT Diels-Alder reaction
(intramol., of allenic dienamide, in total synthesis of trikentrin B)
IT 542-92-7, 1,3-Cyclopentadiene, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(Diels-Alder reaction of, with allenic ester)
IT 16666-78-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(Wittig reaction of, with tetracyclic carboxaldehyde)
IT 2450-71-7, Propargylamine
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation of, with bicycloheptenecarboxaldehyde derivative)
IT 127477-96-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and Diels-Alder reaction of, with cyclopentadiene)
IT 127478-04-0P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and Wittig reaction of, with propylidene triphenylphosphorane)
IT 127477-99-0P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and condensation of, with formaldehyde)
IT 127477-98-9P
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation and condensation of, with propargylamine)
IT 127478-01-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and dehydrogenation of)
IT 127478-02-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and detritylation of)
IT 127477-95-6P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and isomerization of)
IT 127478-05-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and oxidative cleavage of)
IT 127478-03-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and oxidation of)
IT 82816-38-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reaction of, with Et diazoacetate)
IT 127478-06-2P 127515-91-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reduction of)
IT 127478-00-6P 127592-48-7P, (±)-(Z)-cis-Trikentrin B
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 127477-97-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation, reduction, and oxidation of)
IT 127515-90-6P, (±)-Trikentrin B
RL: SPN (Synthetic preparation); PREP (Preparation)
(total synthesis of)
IT 107-19-7, 2-Propyn-1-ol
RL: RCT (Reactant); RACT (Reactant or reagent)
(tritylation of)

RX(90) OF 105 - 11 STEPS



60%

NOTE: 6) Mol. sieves 1st step

L18 ANSWER 7 OF 7 CASREACT COPYRIGHT 2004 ACS on STN
AN 112:217478 CASREACT
TI Synthesis of indoles and L-tryptophans specifically deuterium or carbon-13-labeled in the six-membered ring
AU Van den Berg, Ellen M. M.; Van Liemt, Willem B. S.; Heemskerk, Bram; Lugtenburg, Johan
CS Gorlaeus Lab., Univ. Leiden, Leiden, 2300 RA, Neth.
SO Recueil des Travaux Chimiques des Pays-Bas (1989), 108(9), 304-13
CODEN: RTCPA3; ISSN: 0165-0513
DT Journal
LA English
AB (4-2H)-, (5-2H)-, (6-2H)-, (7-2H), (4-13C)- and (5-13C)-1H-indole and the corresponding six L-tryptophans were prepared from simple labeled starting materials via a single reaction scheme. The L-tryptophans were prepared from the indoles via a quant. one-step biosynthetic conversion. A scheme leading to the preparation of three other indoles, mono-13C-enriched in the 6-membered ring, has been developed and optimized. Indole and L-tryptophan, 13C- or 2H-enriched at positions 4 or 5, were prepared with 99% isotope incorporation without scrambling. (6-2H)-1H-indole and -tryptophan were prepared with 95% D incorporation, and (7-2H)-1H-indole and -tryptophan with 96%. The reactions have been carried out on a gram scale and, in the case of the 4-13C system, they have been scaled up to the 10 g level with no deterioration in yield.
CC 34-2 (Amino Acids, Peptides, and Proteins)
Section cross-reference(s): 27
ST carbon 13 labeled indole tryptophan; deuterium labeled indole tryptophan
IT 107-19-7, Propargyl alcohol
RL: RCT (Reactant); RACT (Reactant or reagent)
(addition reaction of, with dihydropyran)
IT 2450-71-7, Propargyl amine
RL: RCT (Reactant); RACT (Reactant or reagent)
(condensation reactions of, with labeled and unlabeled crotonaldehyde)
IT 56-45-1, Serine, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(enzymic reaction of, with labeled and unlabeled indoles, tryptophans from)
IT 75-07-0, Acetaldehyde, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(olefination of, with triethylphosphonoacetate)
IT 78633-30-4P 127136-18-9P 127136-19-0P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and acidic hydrolysis of)
IT 6089-04-9P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and addition reaction of, with tributyltin hydride)
IT 127135-91-5P 127135-92-6P 127135-93-7P 127774-47-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and condensation of, with formaldehyde, allene derivative from)
IT 127135-90-4P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and condensation of, with labeled and unlabeled formaldehyde, allene derivs. from)
IT 123-73-9P 84041-41-8P 127136-15-6P 127136-16-7P 127136-17-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and condensation of, with propargylamine)
IT 120-72-9P, 1H-Indole, reactions 52199-98-1P, 1H-Indole-5-d

107266-42-2P, 1H-Indole-6-d 127136-06-5P, 1H-Indole-4-d 127136-07-6P, 1H-Indole-7-d 127136-08-7P, 1H-Indole-4-13C 127136-09-8P, 1H-Indole-5-13C
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and enzymic reaction of, with serine, tryptophan from)

IT 764-01-2P, 2-Butyn-1-ol
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and hydride or deuteride reduction of)

IT 623-70-1P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and hydride reduction of)

IT 867-13-0P, Triethylphosphonoacetate
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and olefination by, of acetaldehyde)

IT 504-61-0P 73156-71-5P 119711-86-3P 127136-20-3P 127136-21-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and oxidation of, with manganese dioxide, crotonaldehyde from)

IT 13307-67-0P, 1-Carboethoxyindole 127136-00-9P 127136-01-0P 127136-02-1P 127136-03-2P 127136-04-3P 127136-05-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and saponification of)

IT 55723-10-9P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and sequential lithium-tin exchange and reaction of, with labeled and unlabeled iodomethane)

IT 104837-80-1P 127135-94-8P 127135-95-9P 127135-96-0P 127135-97-1P 127135-98-2P 127135-99-3P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and sequential thermal cyclization and aromatization of)

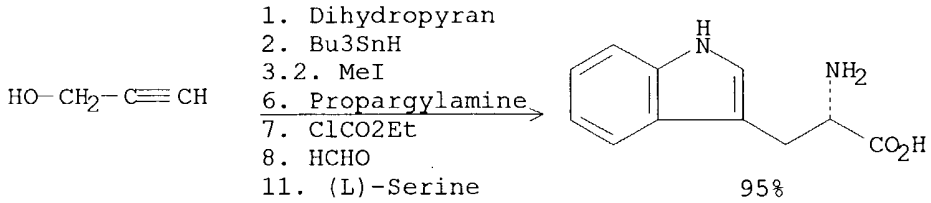
IT 127136-10-1P 127136-11-2P 127136-12-3P 127136-13-4P 127136-14-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and N-acylation of, with Et chloroformate)

IT 87830-21-5P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

IT 73-22-3P, Tryptophan, preparation 90697-20-4P, L-Tryptophan-4-d 105201-56-7P, L-Tryptophan-7-d 127136-22-5P, L-Tryptophan-5-d 127136-23-6P, L-Tryptophan-6-d 127136-24-7P, L-Tryptophan-4-13C 127136-25-8P, L-Tryptophan-5-13C
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of, by enzymic reaction of indole with serine)

IT 105-36-2, Ethyl bromoacetate
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with tri-Et phosphite)

RX(379) OF 408 - 11 STEPS




95%

NOTE: 2) 72% overall, 6) mol. sieves agent, 11) enzymic; microbial; buffered soln.

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